



# Microsample Characterization of Coatings for GRCop-84 for High Heat Flux Applications

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## Project Summary

A multidisciplinary Johns Hopkins University-NASA Glenn team is undertaking a collaborative research program to elucidate and model the thermal stability and mechanical integrity of candidate coatings for GRCop-84. GRCop-84 is a high conductivity, high strength copper alloy that was recently developed at NASA Glenn for use in high temperature, high heat flux applications. With potential applications in rocket motor combustion chamber liners, nozzle ramps and other actively cooled structures, this new material offers great potential for decreasing weight and increasing reliability of third generation reusable launch vehicles. Current emphasis has turned toward the development of environmentally resistant and thermal barrier coatings for this alloy. Metallic coatings such as NiCrAlY and Cu-8-30%Cr have shown promise in: prohibiting blanching, reducing 'dog-house' failures, increasing operating temperatures and decreasing cooling requirements. The focus of this research program is to develop a fundamental understanding of the substrate-coating interactions that occur during thermal cycling (inter-diffusion, viscoplasticity, morphological evolution, crack formation, etc.) and to derive a science-based protocol for future coating selection, optimization and reliability assurance. The microsample tensile testing approach adopted for this study allows us to characterize small-scale and highly scale-specific coatings and properties in a way not possible by conventional means. In addition to providing much needed design data, the integration of microsample testing with detailed microstructural observations provides a mechanistic foundation for coating optimization and life prediction modeling.

## Research Objectives

The overall objective of this project is to combine microsample testing, and detailed microstructural observations in a manner that will elucidate the thermal cyclic behavior and overall performance of candidate coatings for GRCop-84. The specific tasks for the project are outlined below. Tasks 1-4 were listed in the original proposal. Difficulty associated with obtaining plasma sprayed coatings from the NASA subcontractors has meant that NASA was unable to supply JHU with the candidate coatings in the first year of this study. Consultation between NASA and JHU has resulted in the following plan of action. Tasks 1-4 have been slightly modified and shifted to years 2 and 3 of the collaboration. Specific details related to these tasks are addressed in the section entitled Plans for Future Work. An additional task (5) has been added and was conducted in the first year of the study, and the experimental results and discussion associated with this portion of the project are given in the next section.

1. *NASA to deposit three candidate coatings on GRCop-84 buttons. Samples to be provided to JHU in the following conditions: as deposited, after 50 thermal cycles and after 100 cycles.*
2. *JHU to cut and prepare microsamples from the coatings and measure the temperature dependent CTE, Young's modulus, tensile strength and creep strength of each coating.*
3. *JHU to conduct microstructural characterization of as-processed and cycled coatings to document microstructural evolution and discern high-temperature deformation and failure mechanisms.*

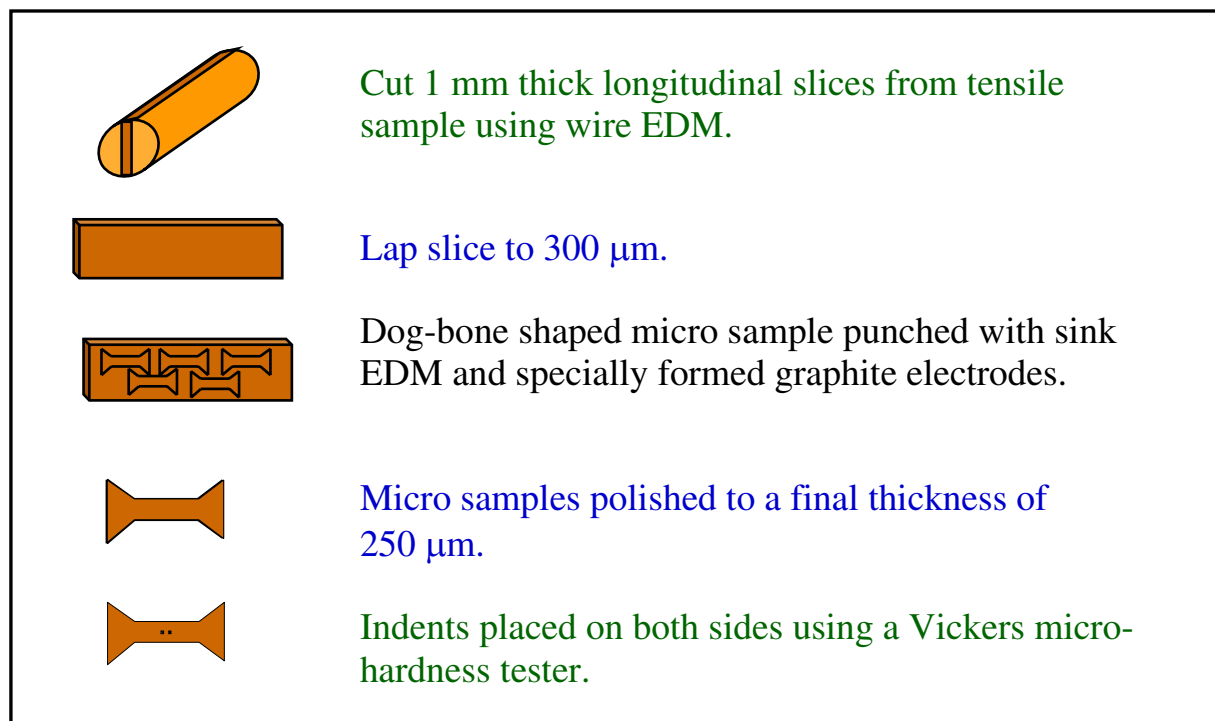
4. *JHU and NASA will incorporate the microsample data into FE-based life cycle models that emphasize the interplay of the high temperature gradients, thermally induced strains and elevated temperature mechanical properties.*
5. *JHU to prepare and test microsamples from extruded and rolled sheets of GRCop-84. The results of these microsample tests are to be compared with macro sample experiments conducted at Glenn Research Center and used to characterize the mechanical response of the alloys in various process states.*

## Experimental Results and Discussion

**Specimen preparation.** GRCop-84 was provided to Hopkins in three different forms:

1. A macro tensile sample that had been machined from an extruded bar
2. A cross-rolled sheet that had been rolled to a thickness of 40 mils and heat treated at 400°C for 15 minutes and
3. A 20 mil cross-rolled sheet that received no post roll heat treatment

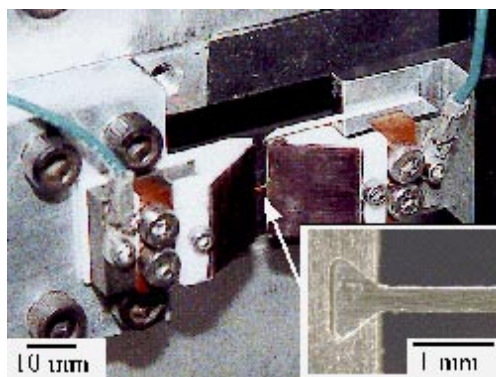
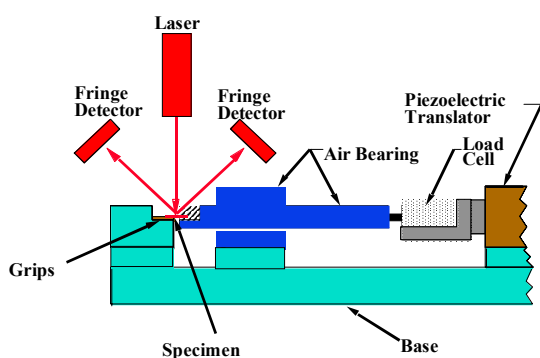
Microsample were prepared from each of these three conditions. The extruded tensile sample was sliced along the longitudinal axis with a wire EDM, lapped to a thickness of 300 microns and microsamples were punched from these sheets using a plunge EDM and a specially shaped graphite electrode, see Fig. 1. The front and back surfaces were polished to a mirror surface with a tripod polisher and Vickers microhardness indents were placed on each side of the specimens.



**Fig. 1:** Schematic of steps for microsample preparation from extruded GRCop-84 samples.

The indents act as reflective markers for the interferometric strain/displacement gage (ISDG). The 20 and 40 mils sheets of GRCop-84 were prepared by using the last four steps in Fig. 1.

**Microsample testing.** The samples were tested in the Hopkins microsample tensile machine. A schematic and photograph of the JHU microsample tester are shown in Fig. 2. Self-aligning grips, which match the ends of the bowtie shaped specimens, hold the specimens in place while a linear air bearing insures proper alignment of the load frame, greatly reduces friction in the loading mechanism, and permits accurate measurement of the load with an in-line miniature load cell. Loading was accomplished through the use of a piezoelectric actuator and a newly purchased micro-actuator screw drive. The microsample tensile tests included in this report were conducted at a constant strain rate of  $1 \times 10^{-4}$  per second in air at room temperature.



**Fig. 2:** Schematic of the microsample testing machine and photo of the gripping arrangement employed for high temperature testing.

**Results for extruded GRCop-84.** Two tensile curves that were obtained using microsamples of extruded GRCop-84 are shown in Fig. 3(a). These microsamples were loaded into the plastic regime and then unloaded and reloaded to allow for measurements of the Young's modulus. The microsamples were subsequently pulled to failure. The strains to failure measured directly on the specimen (~12 to 14%) were found to be very close to the values given by the ISDG. The similarity of the two curves is also a strong indication of the repeatability of the microsample experiments. Two macrosample curves that were provided to us by colleagues at NASA Glenn are shown in Fig 3(b). Comparison of these suggests that there is good agreement between the microsample and macrosample experiments. This point was further verified by combining all four curves on the same plot. Measured values for the Young's modulus and yield strength are included in this figure. The close correlation between the two techniques was taken as a strong verification of the microsample technique, which has also been used to successfully measure the mechanical response of a wide range of materials, including but not limited to: polysilicon thin films [1,2]; base metal, recast and heat affected zones of steel weldments [3,4]; LIGA Ni, Cu and Permalloy [5-7]; SU-8 photo-resist [8]; single crystalline TiAl [9-11]; fully-lamellar and polysynthetically twinned TiAl based alloys [12,13]; nanocrystalline Ni, Cu and Al [14-17]; and platinum modified nickel aluminide bond coats for thermal barrier coatings [18-20].

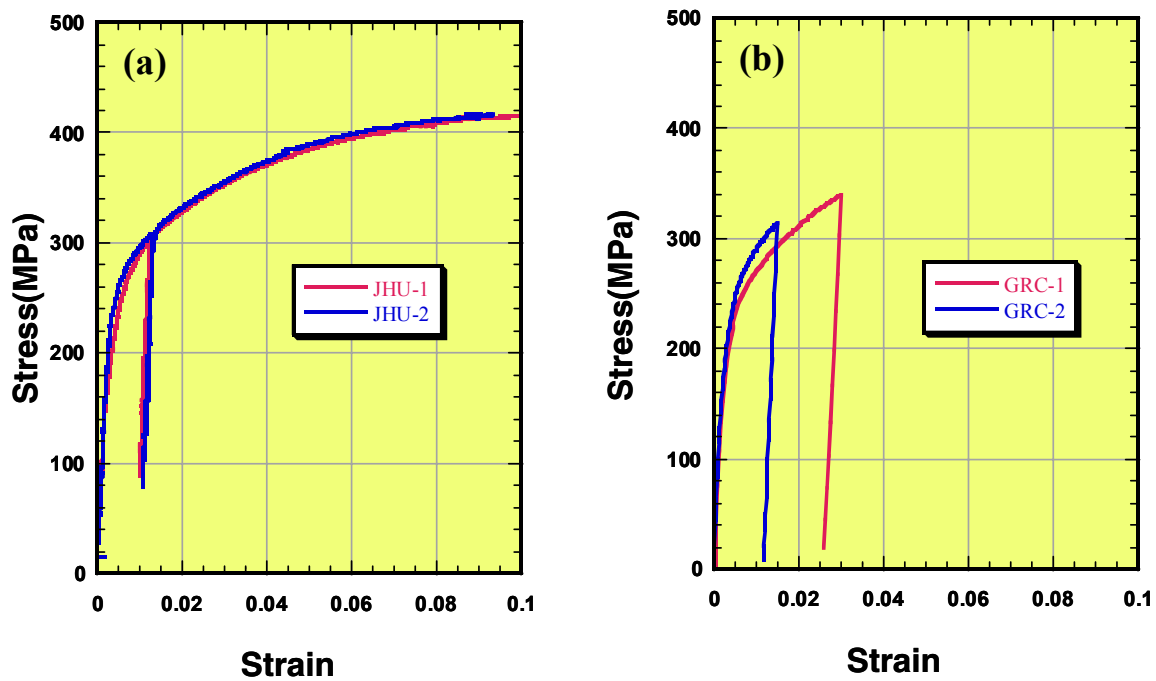


Fig. 3: (a) JHU microsample tensile curves of extruded GRCop-84, and (b) GRC macrosample tensile tests of the same material.

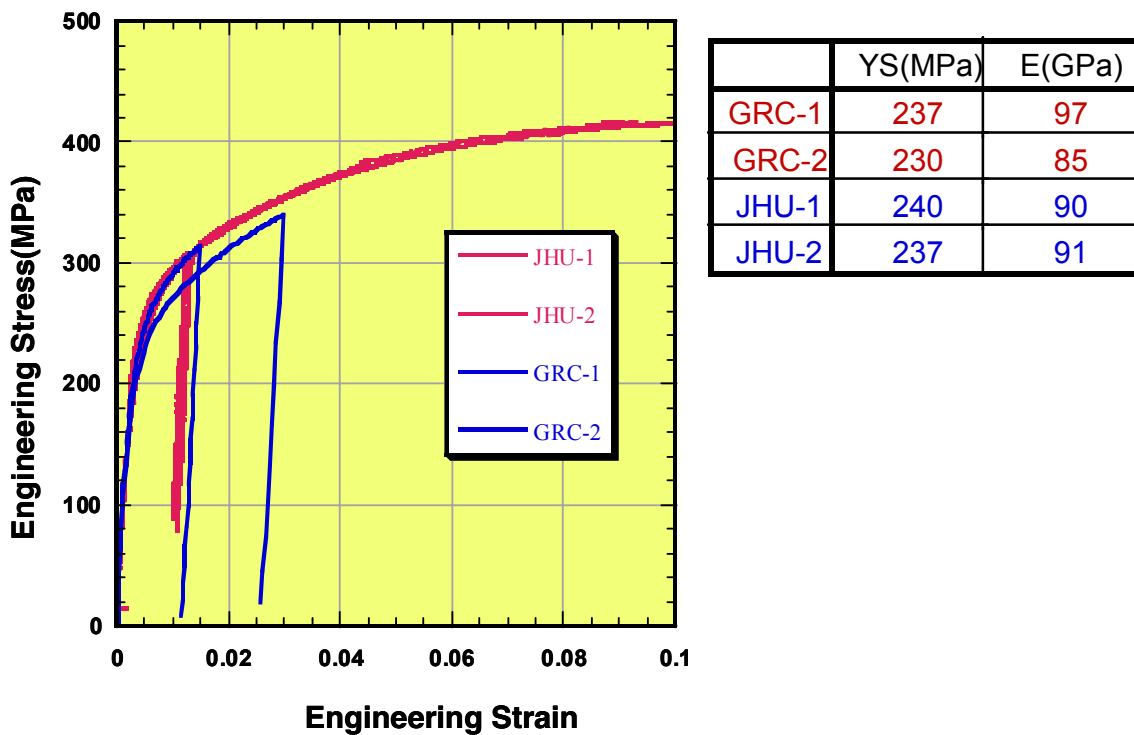


Fig. 4: Direct comparison of the microsample and macro sample curves in Fig. 3.



**Results for cross-rolled GRCop-84.** Microsample tensile tests for the cross-rolled specimens are shown in Fig. 5. A composite curve of microsample curves for each of the three conditions, extruded, cross-rolled to 40 mils and annealed, and cross-rolled to 20 mils with no anneal, are shown in Fig. 6. Comparison of these curves indicates the expected trends, e.g., that increased amount of cold/warm work leads to increased strength and decreased ductility. The microsample tests conducted in the first year have allowed us to verify the validity of the technique and to characterize GRCop-84 in various processing states. Further characterization of the GRCop-84 alloy can and will be done using macrosamples. The true potential of the microsample tests lies in their ability to characterize thin films and coatings and that will be the focus of the remaining years of this project.

**Elevated temperature testing.** Efforts at JHU are currently focused on obtaining elevated temperature (600°C) microsample tensile and creep tests. Elevated temperature microsample testing is accomplished by resistively heating them with a DC current. As a result of their extremely small cross-sections, the heating is concentrated in the gage and temperatures in excess of 1500°C have been achieved. A two-color infrared pyrometer with a 290  $\mu\text{m}$  spot size is used to measure temperature directly on the sample surface during testing with 1°C resolution. Ultra-fine 12  $\mu\text{m}$  thermocouples attached to the gage section of dummy microsamples have been used to ensure proper calibration of the pyrometer, and temperature profiles taken during testing have shown that the temperature gradient is less than 5°C over the gage of the microsample. Elevated temperature tests of platinum modified nickel aluminide bond coats have been successfully conducted in air see for example [19,20]. However, the presence of the GRCop-84 substrate in the present study will require that controlled atmosphere experiments be conducted. We have built a transparent chamber for our microsample tester that will allow us to test under a controlled argon atmosphere while still using the optical laser based ISDG strain measurement system, and we will add controlled atmosphere high temperature microsample testing to our repertoire in the early part of FY03.

## Plans for Future Work

**Coatings to be tested.** The general theme of the proposed research will remain the same for the remainder of the project, but the exact number and composition of the coatings to be tested will be varied from the original proposal in accordance with discussions with Dr. Sai Raj at NASA Glenn. New coatings are still being developed in this project and JHU is committed to testing the coatings that are most relevant to the NASA program. To that end, NASA has obtained plasma sprayed monolithic coatings of Cu-8Cr, Cu-26Cr, NiCrAlY and NiAl coatings, and JHU will conduct a limited number of microsample tests and obtain RT and 600°C tensile curves for the Cu-26Cr and NiCrAlY coatings in the early part of FY03. NASA is also obtaining and will provide plasma sprayed overlay coatings of NiCrAlY and Cu-XCr (where X will range from 8 to 56). These overlay coatings will be provided to JHU on the GRCop-84 substrate, and they will be tested in both the as-deposited and thermally cycled conditions. The effects of coating chemistry and thermally cycling will be assessed with the two types of microsample tests that are described below. These tensile tests will be paralleled by extensive TEM investigations of the underlying microstructure as described in the original proposal.

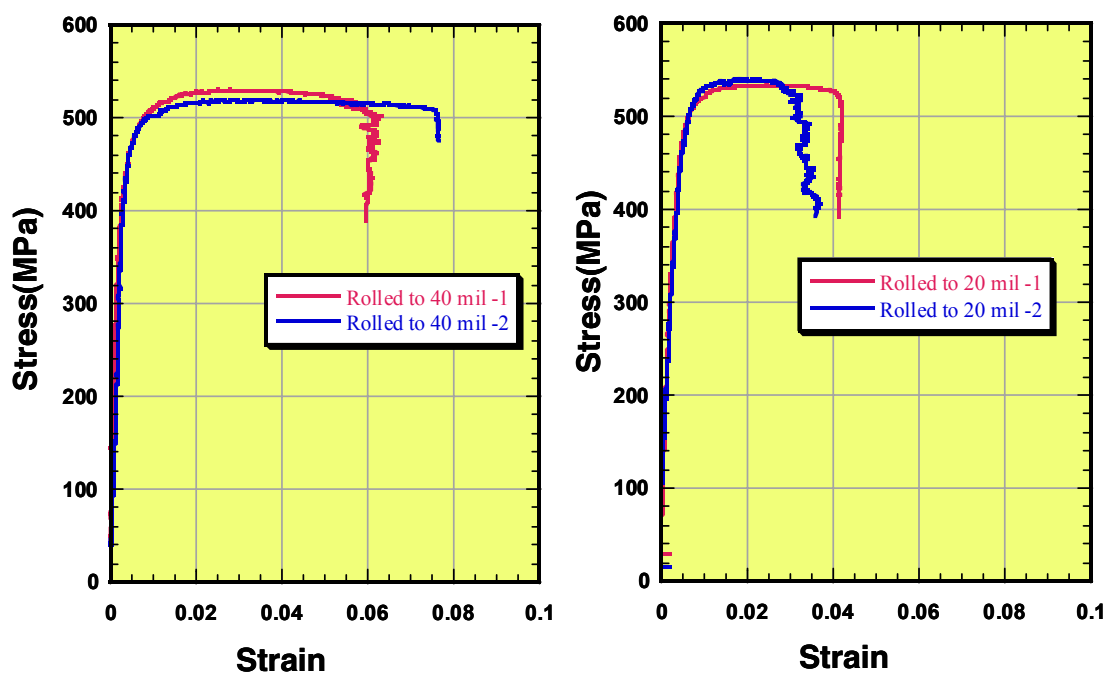


Fig. 5: Microsample tensile curves for cross-rolled GRCop-84.

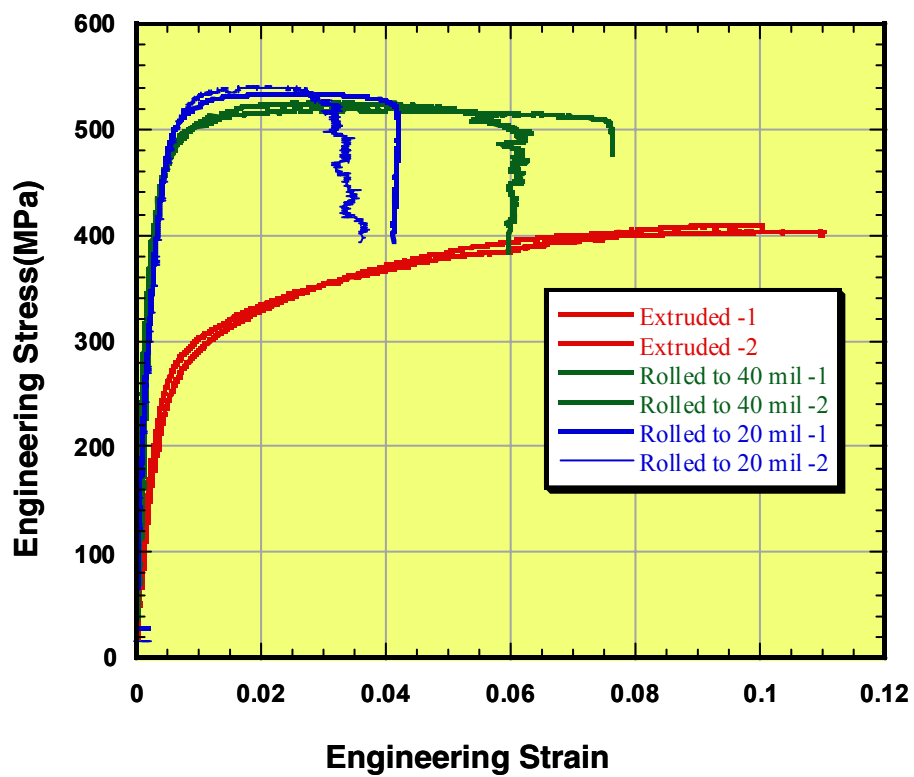
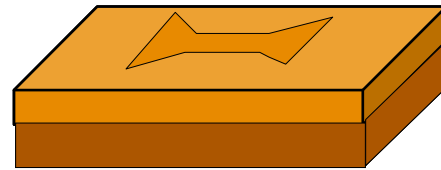


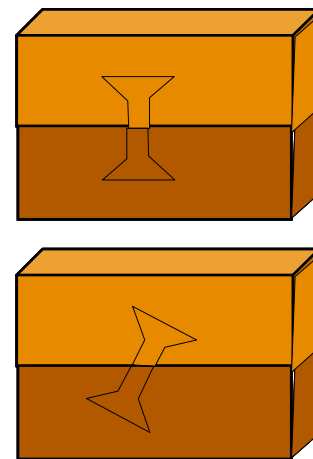
Fig. 6: Composite of all microsample tensile curves.

**Coating properties experiments.** In-plane tensile and stress relaxation microsample experiments provide a unique opportunity to measure the small scale and highly scale specific properties of as-deposited and thermal cycled coatings. Here we will focus on two coatings, Cu-26Cr and NiCrAlY that have been plasma sprayed (at PPI) onto 1 in. diameter GRCop-84 coupons. Four coupons of each material will allow for tensile and creep testing of as-deposited and two thermal cycled conditions, as well as microstructural characterization. After thermal cycling and before testing the substrate will be removed and the microsample cut in the in-plane orientation, as is shown in Fig. 7.



**Fig. 7:** In-plane geometry for testing of overlay coatings with thickness of ~100 microns.

**Bond strength experiments.** The following experiments were derived and added to the program during a research meeting at NASA Glenn in September of 2002. The new experiments will involve preparation and testing of microsamples with the GRCop-84/coating interface perpendicular and at an angle of 45° to the tensile axis. These experiments will be relatively easy for us to conduct as they will not require ISDG strain measurement and will provide much needed interfacial strength information for modeling. The GRCop-84 substrate and overlay coatings will both have to be 3 mm thick to allow for preparation of the cross-wise microsamples. Measurements will be made for: two coatings (Cu-26Cr and NiCrAlY), 3 surface conditions (polished, light and heavily grit blasted), and 3 states (as-deposited and 2 thermal cycles). One-inch square coupons of each coating will be sliced and the slices thermal cycled (or not) to get the 3 different states.



**Fig. 8:** Out-of-plane geometry for testing the bond strength of overlay coatings for various processing and thermal cyclic conditions.

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## Publications

None yet



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